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(GASMAN)

PRINCIPAL INVESTIGATOR: Dr. Jamine Lee

CONTRACTING ORGANIZATION: Spectral Sciences, Inc.
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The U. S. Army requires an instrument for monitoring gaseous by-products, including HCl, NO₂, CO₂, CO, etc. generated by newly developed gunpowder and combustion fuels, and in evaluating their threats to the health of soldiers. It is desired to perform simultaneous real time monitoring of these gas concentrations in ambient air with detection sensitivities at or below the Threshold Limit Value (TLV) levels. This Phase II SBIR final report discusses the features and capabilities of the Spectral Sciences, Inc. (SSI) Gallium Arsenide Semiconductor-laser Multigas Analyzer (GASMAN) for detecting multiple toxic gases using optical absorption. GASMAN utilizes commercial indium gallium arsenide phosphate (InGaAsP) diode lasers developed for communications and digital storage and readout. The high reliability and stability of these lasers make them well-suited for instruments requiring long-term unattended operation. Line-locking and laser wavelength modulation techniques implemented in SSI's field instrument designs insure selective, sensitive detection of the desired species on a time scale of 80 ms.

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Principal Investigator's Signature

Sept. 20, 96
Date

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EXECUTIVE SUMMARY

The Spectral Sciences, Inc. (SSI) Gallium Arsenide Semiconductor-Laser Multigas Analyzer (GASMAN) is a portable and rugged diode laser-based instrument for automated, real-time monitoring of CO, CO₂, HCl, NO₂, and other gaseous air pollutants during U. S. Army field operations. By monitoring these pollutants, which are generated from gunpowder, rocket propellants, and other ordnance, GASMAN will significantly enhance Army capabilities for assessing the health impact of weapons systems on field personnel.

GASMAN incorporates two interchangeable laser modules to measure two different gas species simultaneously. The instrument is enclosed in a weatherproof 18" x 12" x 14" carrying case with a top handle and shoulder strap attachments and weighs approximately 50 lbs. A total of three laser modules have been developed and are supplied with the instrument: (1) a module for CO or CO₂, (2) a module for HCl, and (3) a module for NO₂. Additional modules can be provided by SSI for other gas species.

Key features of GASMAN include a very wide dynamic range (10^4), very fast response (less than 0.1 second), flexible, programmable data acquisition and capabilities for communication and control with external devices. GASMAN provides both real-time concentration readout and data logging capability. The data sampling rate is adjustable from 0.08 s to 300 s per measurement; 10 Mbyte of memory are provided to permit collection and storage of hours to months of data during unattended operation.

In addition to Army and other U.S. Defense service applications, there are a variety of potential commercial uses in the general areas of research, regulatory compliance, and industrial plant on-line process monitoring and control.

All of the technical goals of the Phase II program were met and the instrument performed as anticipated. This Final Technical Report is part of the deliverable GASMAN documentation and summarizes the instrument design and test results.

1. INTRODUCTION

The U. S. Army requires an instrument for monitoring gaseous by-products, including HCl, NO₂, CO₂, CO, etc. generated by newly developed gunpowder and combustion fuels, and in evaluating their threats to the health of soldiers. It is desired to perform simultaneous real time monitoring of these gas concentrations in ambient air with detection sensitivities at or below the Threshold Limit Value (TLV) levels.

This Phase II SBIR final report discusses the features and capabilities of the Spectral Sciences, Inc. (SSI) Gallium Arsenide Semiconductor-laser Multigas Analyzer (GASMAN) for detecting multiple toxic gases using optical absorption. GASMAN utilizes commercial indium gallium arsenide phosphate (InGaAsP) diode lasers developed for communications and digital storage and readout. The high reliability and stability of these lasers make them well-suited for instruments requiring long-term, unattended operation. Line-locking and laser wavelength modulation techniques⁽¹⁻⁵⁾ implemented in SSI's field instrument designs insure selective, sensitive detection of the desired species on a time scale of 80 ms. The basic features of the approach are discussed below. The detailed instrument layout, the setup, operation, maintenance and laser module replacement procedures, and a description of the software are found in the *User's Manual*.

2. TECHNICAL APPROACH

2.1 Basic Concept

The spectroscopic method for gas measurement used by GASMAN takes advantage of the fact that the molecular absorption lines are narrow compared to the average spectral interval between the lines. Thus, it is usually possible to identify one or more lines of the species to be measured that are well-separated from lines of other interfering species occurring in the same wavelength region. The measurement is performed by passing monochromatic near-infrared light from a thermoelectrically-cooled GaAs-based diode laser through the sample cell. The sample cell in GASMAN contains Herriott multipass optics which provide an absorption path length of approximately 11 m.

The sensor relies on two techniques of diode laser spectroscopy, harmonic signal detection and wavelength stabilization using line locking.⁽¹⁻⁵⁾ The laser wavelength and intensity are modulated at a frequency f by applying a sinusoidally varying current to the laser. Harmonic signals are extracted by phase-sensitive amplifiers at integer multiples of the modulation frequency (i.e., $1f$, $2f$, $3f$, etc.). These signals are used to determine the transmitted light intensity, the strength of the molecular absorption line, and the displacement, if any, of the center wavelength from the center of the line.

Each order of harmonic signal is directly related to, but not identical to, the corresponding order of the derivative of the line shape with respect to the wavelength. The second harmonic ($2f$) signal is proportional to the absorptivity. The transmitted light intensity, as measured by either the $1f$ or DC signals, is used for normalization. The mathematical relationship between the normalized $2f$ signal and the absorptivity at the line center has been previously derived.⁽¹⁻⁵⁾ The third harmonic ($3f$) signal is acquired from a separate beam that passes through a cell filled with a reference gas, such as the one to be detected. Since the $3f$ signal is proportional to the displacement from the line center for small displacements, it provides an error signal for stabilizing the center wavelength to the center of the line (line-locking).

In GASMAN, line-locking begins automatically at startup. The starting laser operating temperature and current are set to tune the laser close to the absorption line when it is first turned

on. The line-lock servo amplifier generates an error signal which is proportional to the $3f$ signal averaged over many seconds. After a specified warmup period, the error signal is applied to the current controller, driving the $3f$ signal to zero and bringing the center wavelength into coincidence with the line center.

2.2 Design Features

GASMAN incorporates two interchangeable diode laser modules for measurements of gas species concentrations (Figure 1). Each laser wavelength is sinusoidally modulated at a frequency f ; the center wavelength coincides with a molecular absorption line of the species to be measured. The laser beam passes through a multiple-pass optical cell containing the sample and falls on a photodiode (Figure 2). The $2f$ signal, which is proportional to the absorptivity, is acquired using analog lock-in electronics and is converted to species concentration using a single-board computer as a digital signal processor. The concentration values are stored in the on-board flash memory, displayed on an LCD, and output to external devices such as a PC via a serial port.

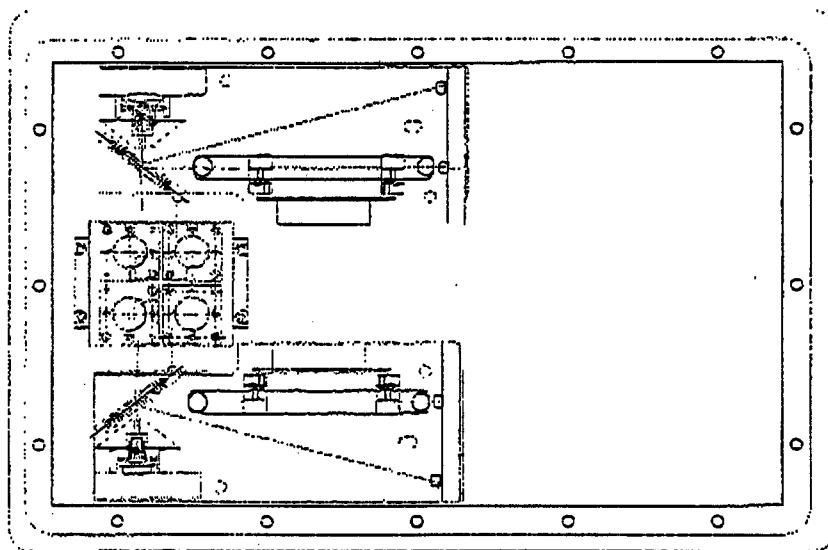


Figure 1. Laser Modules on Mounting Plate. Steering Mirrors are Between the Modules, and Dashed Lines Indicate the Beams.

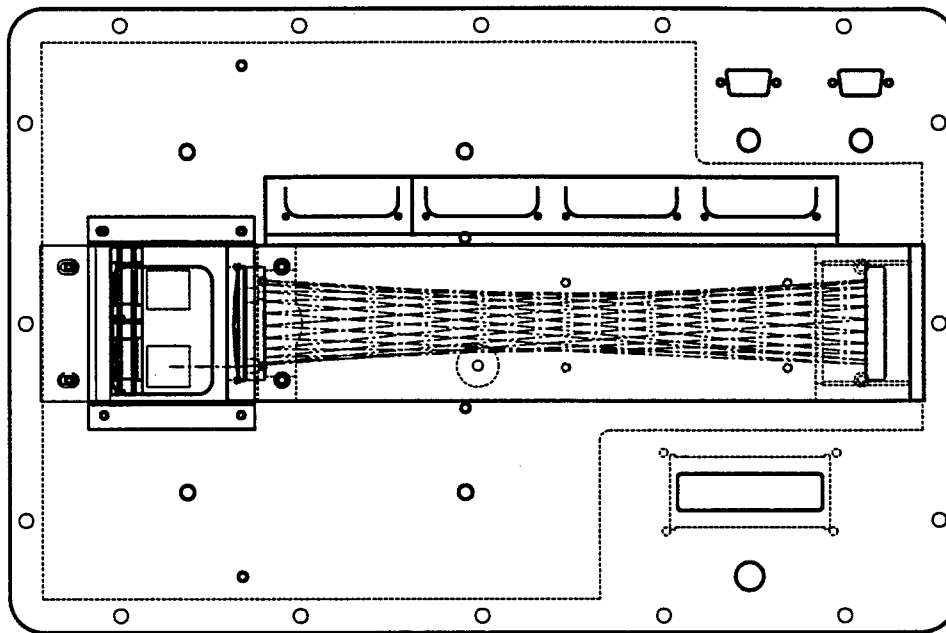


Figure 2. Multipass Sample Chamber.

To keep the laser wavelength centered on the molecular absorption line, an optical path through a reference cell filled with the gas species to be measured is provided in each laser module assembly (Figure 1). The $3f$ absorption signal from the reference path photodiode is acquired and incorporated in a feedback loop driving the laser. In addition, a third photodiode provides a null (zero-absorption) $2f$ signal that is subtracted from the sample signal. Further details of this $2f$ -detection, $3f$ -line-locking method are given in several papers.⁽¹⁻⁵⁾

Features and specifications of the sensor are:

- Dimensions and Weight: 18" x 12" x 14", 50 lbs
- Open-Air Sampling with Built-in Fan
- Operating Temperature Range: 0 to 60 °C
- +12 or +24 VDC Battery Power Input
- Power Consumption @+12VDC: 0.75 A with laser off, 6 A at startup, 3 A average with laser on
- ppm Concentration Displays for 2 Simultaneous Gas Species
- On-Board Data Logging with 10 Mbyte Flash Memory
- TLV Sensitivity, Low Cross-Interference, and 10^4 Dynamic range

- Plug-in Species Modules and User-Selected Sampling Rate (0.08 to 300 sec)
- Laser Modules Include NO₂, CO₂/CO and HCl
- Remote Control Through RS232 or RS422 Serial Communication (19200, 8, 1, n)
- Choice of Automated or User-Interactive Operating Modes

3. PERFORMANCE CHARACTERIZATION

3.1 Laser Module Characteristics

Three delivered laser modules have been characterized during the Phase II program. The 1.57 micron laser used for monitoring CO₂ and CO is a DFB laser, while the 1.21 micron and 0.67 micron lasers used for monitoring HCl and NO₂ are multimode Fabry-Perot lasers. Their specifications and spectroscopic parameters are summarized in Table 1.

Table 1. Laser Characteristics for the Three Delivered Laser Modules.

Target Gas	CO ₂ /CO	HCl	NO ₂
Manufacturer	GEC Marconi	Mitsubishi	Toshiba
Part Number	9419N8	ML8701 ^a	TOLD9215(s)
i _{th} (mA)	35	15	35
i _{op} (mA)	250	30	45
dω/dT (cm ⁻¹ /°C)	-0.329	-0.528	-2.04
dω/di (cm ⁻¹ /mA)	-0.028	-0.045	(-0.386) ^c
dω/di (@3kHz)	-0.016	-0.02	(-0.386) ^c
SBR (dB)	-36	NA ^b	NA ^b
Tuning Range (cm ⁻¹)	6329 - 6344	8277 - 8356	14785 - 14885
Power (mW)	50	5	10

^a discontinued

^b not applicable for a Fabry-Perot laser

^c estimated

For each species, an absorption line accessible by current- and temperature-tuning the laser was selected to minimized ambient gas interferences such as CO₂ and water. The laser parameters for operating at these wavelengths are summarized in Table 2. Table 3 lists the spectroscopic parameters for the lines from the HITRAN compilation.⁽⁶⁾

Table 2. Laser Operating Parameters.

Target Gas	CO ₂	CO	HCl	NO ₂
T _{LD} (°C)	25.21	37.95	30.28	19.35
i _{LD} (mA)	162.23	247.67	25.04	34.86
ω (cm ⁻¹)	6336.24	6330.17	8278.75	(14858) ^a
A _{modulation} (cm ⁻¹)	0.129	0.129	0.136	(0.248) ^a
I _o ^{smp} (V)	2.86	4.19	0.7	-1.33

^a estimated values

Table 3. Molecular Line Parameters.

Gas	CO ₂	CO	HCl
S (x10 ⁻²³ cm/molec)	1.596	1.589	5.24
γ _{air} (cm ⁻¹ /atm)	0.0754	0.0654	0.0724
γ _{self} (cm ⁻¹ /atm)	0.1043	0.0719	0.2529
E (cm ⁻¹)	81.94	57.67	125.21
n	0.69	0.69	0.71

3.2 Sensor Calibration

For the three delivered laser modules, the instrument response using these molecular lines was calibrated against gas mixtures of 980 ppmv CO₂, 290 ppmv CO, 540 ppmv HCl, and 7 ppmv NO₂ in nitrogen diluent. During the calibration, the fan attachment was removed from the sensor, and the sample volume was enclosed by two side plates with gasket seals and tube fittings. The gas mixture was introduced through one port and vented out the other port into a fume hood. The optical signals were recorded at a rate of 0.5 Hz and conversion factors were defined to scale the output to yield the known mixture concentration. The sample volume was purged with dry nitrogen several times during the calibration to establish the signal baseline.

Figures 3 and 4 show the calibrations for the species pairs CO/HCl and CO₂/NO₂, respectively. In Figure 3, the sample chamber was initially filled with dry nitrogen. The 540 ppm HCl mixture was flowed into the chamber at 250 seconds, and the flow was turned off at

400 seconds. The 290 ppm CO mixture was introduced at 420 seconds, followed by dry nitrogen at 550 seconds. The sequence was then repeated: HCl was flowed in at 750 sec and turned off at 960 seconds, CO was flowed in at 1000 sec and dry nitrogen was flowed in at 1350 seconds. Conversion factors ("ConcPrefactor" in SETMGWD.*nnn* parameter input file) of 11483.12 for CO and 15346.55 for HCl were used to convert the normalized 2f signals into ppm readings, and yield 283 ± 10 and 524 ± 2 ppm for the CO and HCl concentrations, respectively. The baseline for the HCl channel is -0.3 ± 1.1 ppm. The baseline for the CO channel is sensitive to sample pressure variations, and increased from 0.9 to 21.9 ppm during the calibration run.

In Figure 4, where the signal trace for the NO₂ channel is multiplied by 100, the sample chamber was first filled with dry nitrogen. The 7 ppm NO₂ mixture was flowed into the chamber at 240 sec; the sample was switched to the 980 ppm CO₂ mixture at 360 seconds, and was switched back to NO₂ at 750 seconds. Finally, the chamber was purged with dry nitrogen at 1180 seconds. Conversion factors of 14848.48 and 1489.36 per ppm for CO₂ and NO₂, respectively, yield 1024 ± 9 and 8.3 ± 0.1 ppm for the measured CO₂ and NO₂ concentrations. The baseline for the NO₂ channel, which is insensitive to pressure, is -1.7 ± 0.4 ppm. The baseline for the CO₂ channel has a pressure variation similar to the CO channel and an additional variation of ± 13 ppm; it was not zeroed prior to the calibration.

The relatively large drift of the CO₂/CO laser module with sample pressure and over time is due primarily to the combination of beam drift and interference (etalon) effects. Drifts in the beam profile affect the sample and null channels differently and thus decrease the accuracy with which interference patterns or other background signals are subtracted (see the 1st Quarterly Report and the *User's Manual*). Similar background-subtraction problems can also be brought on by thermal- or pressure-induced stresses.

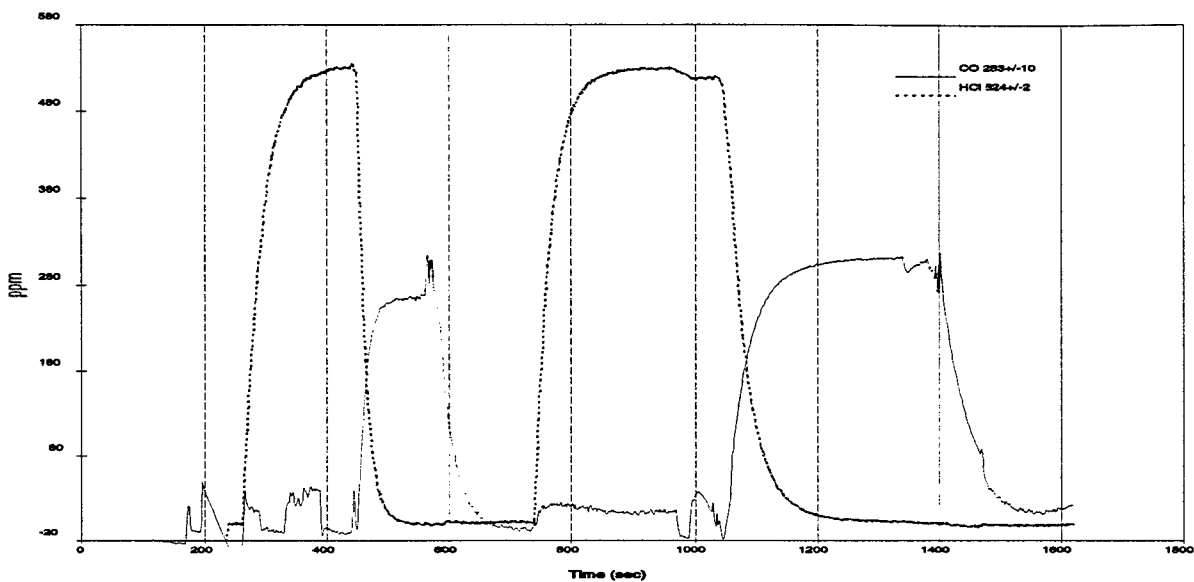


Figure 3. Concentration Calibration Using 290 ppmv of CO (solid line) and 540 ppmv of HCl (dotted line) in nitrogen diluent.

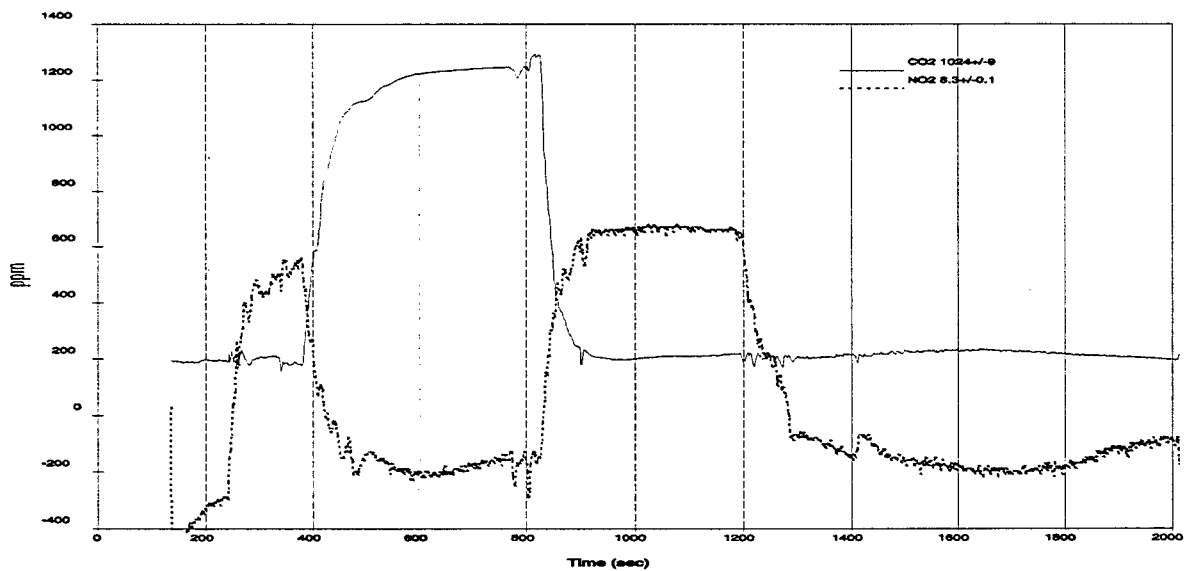


Figure 4. Concentration Calibration Using 980 ppmv of CO₂ and 7 ppm of NO₂. The ppm scale for the NO₂ trace has been multiplied by 100.

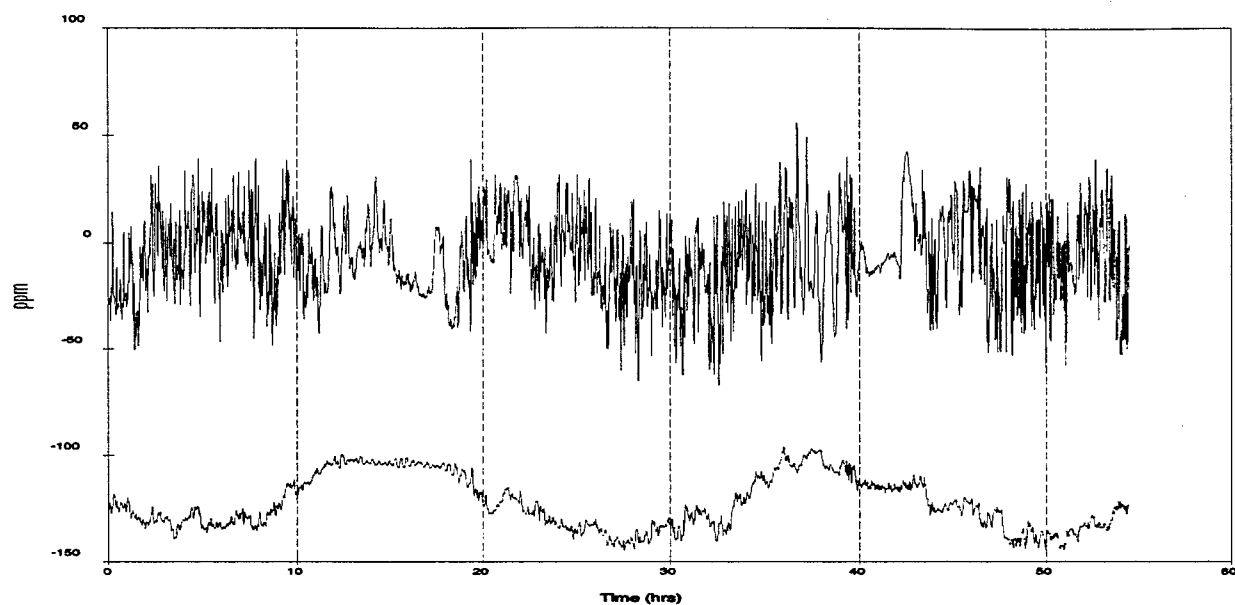


Figure 5a. Stability Testing of the CO and HCl Laser Modules Over 55 Hours: CO and HCl Responses.

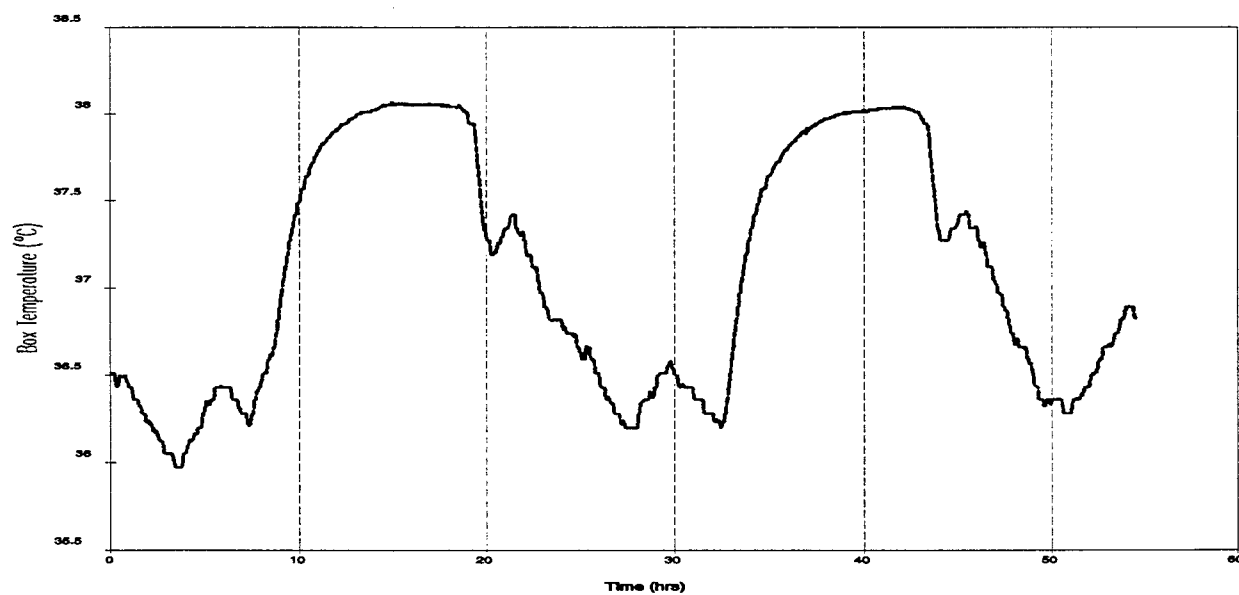


Figure 5b. Stability Testing of the CO and HCl Laser Modules Over 55 Hours: Sensor Temperature.

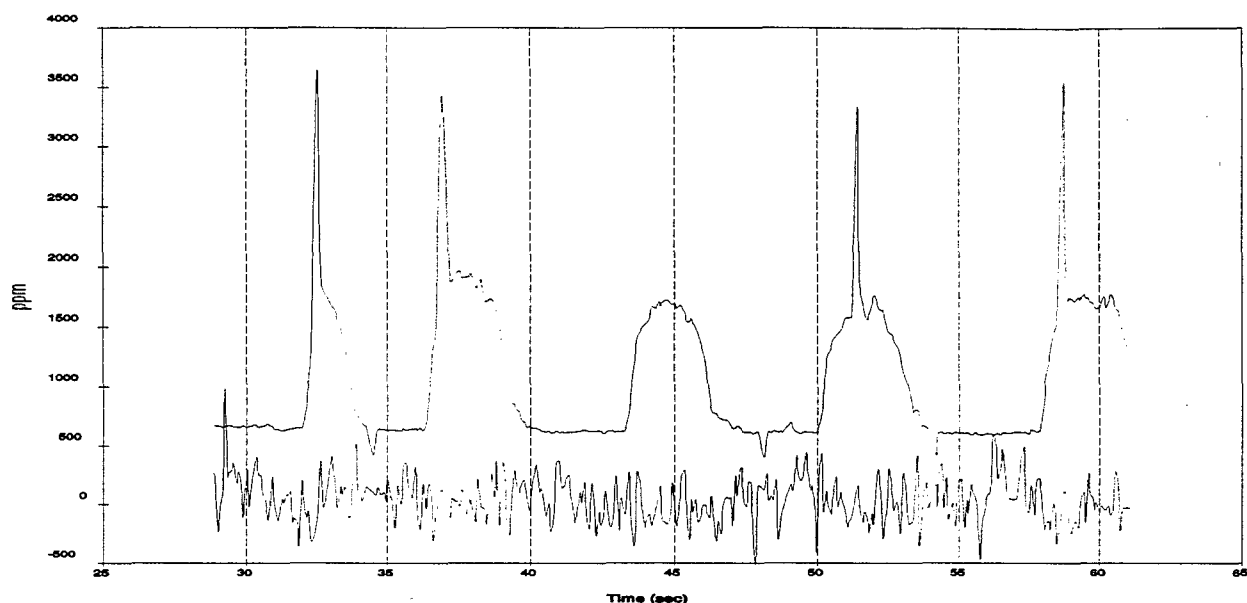


Figure 6. Time Response Test of the CO₂ and HCl Laser Modules. The ppm Scale for HCl Has Been Multiplied by 10.

3.3 Daily Stability

The GASMAN sensor has been tested over several-day periods of continuous operation, in anticipation of long-term, unattended use in the field. Results are shown in Figure 5a for a 55-hour laboratory test run using the CO and HCl laser modules; the data were recorded every 30 seconds. The ppm scale for the HCl laser module is offset by -122.4 ppm for comparison. The sensor ("box") temperature varied between 36 and 38 °C. Since ambient CO and HCl levels are well below ppm levels, the observed fluctuations indicate the sensor stability. The long-term stabilities for these two laser modules are similar, ± 19.3 ppm and ± 12.6 ppm for CO and HCl, respectively. On the other hand, the short-term stabilities over couple of hours are different for these modules, ± 12.7 ppm for CO and ± 3.2 ppm for HCl. The greater stability of the HCl module is attributed to the larger absorption strength of the HCl line (Table 3) and the poorer beam profile of the CO laser module. It should be noted that the stability improved between 14 and 19 hrs and between 38 and 43 hrs when the box temperature was relatively constant. Similar performance is shown in Figures 4a and 4b of the 7th quarterly report for the CO₂ and HCl laser modules.

3.4 Time Response: CO₂ in Human Breath

GASMAN is designed to capture fast transient events. The data acquisition rate is as fast as 100 Hz, and eight data points are averaged and recorded in the on-board flash memory. Figure 6 shows an example of monitoring the ambient HCl (x10 ppm) and CO₂ exhaled by the operator near the sensor. The experiment starts at 28 sec and ends at 61 seconds. Five breathing cycles occur within this time, during which the CO₂ level varied from an ambient level of 628 ± 13 ppm to 3500 ppm at the peak. The sharp rise and fall times indicate fast, efficient sampling by the sensor. The HCl channel remained constant to within $+4.4 \pm 18.8$ ppm, consistent with the HCl detection sensitivity.

4. FUTURE DIRECTIONS

SSI's GASMAN sensor possesses both the performance and features required by the U.S. Army for monitoring gaseous by-products, including CO₂, CO, HCl and NO₂, generated by newly developed gunpowder and combustion fuels, and for evaluating their threats to the health of soldiers. The sensor can be manufactured in quantity with minimal design changes, and additional laser modules can be provided as required. In addition, the software can be upgraded to allow near-simultaneous monitoring of multiple gases with one laser module. These possibilities are elaborated in the following subsections.

4.1 Additional Laser Modules

The GASMAN sensor allows insertion of different laser modules for monitoring different gas species. Three modules for monitoring CO₂/CO, HCl and NO₂ were delivered with the Phase II sensor. Other laser modules can be provided for monitoring different gas species or the same gas species with higher sensitivity. Modules that can be built using available lasers are listed in Table 4.

4.2 Software Upgrade

The use of C source code and a single board computer (SBC) allows the development of simple software upgrades for GASMAN. The next-generation laser diode driver currently being developed by SSI will permit near-simultaneous measurements of multiple gases to be conducted with a single laser module and also provides improved accuracy and background-subtraction capability. A list of gas combinations that could be monitored with a single module is given in Table 5.

Table 4. Available Laser Modules.

Molecule	Laser (nm)	S (cm/molec)	Detection Limit^{a,b}	Acute Limit^b	Threshold Limit^b
NO ₂ ^{c,d}	670 ^e	10 ⁻²¹	0.08	200	3
NO ₂	750 ^e	10 ⁻²²	2	200	3
HCl ^{c,d}	1200 ^f	10 ⁻²²	5	100	5
HF	1313 ^f	10 ⁻²¹	0.16	50	3
HBr	1340 ^f	10 ⁻²³	15	-	3
H ₂ S	1340 ^f	10 ⁻²⁴	75	150	20
HCN	1538 ^f	10 ⁻²¹	0.3	800	10
HI	1538 ^f	10 ⁻²²	5	-	-
NH ₃	1538 ^f	10 ⁻²²	1.5	-	25
CO ^d	1578 ^f	10 ⁻²³	15	1500	35
CO ₂ ^d	1578 ^f	10 ⁻²³	15	100,000	5,000
H ₂ S	1578 ^f	10 ⁻²³	8	150	20
NO	1800 ^f	10 ⁻²³	15	100	25
HCl	1800 ^f	10 ⁻²³	0.05	100	5
H ₂ S	1900 ^f	10 ⁻²³	7.5	150	20
CO ₂	2000 ^f	10 ⁻²²	0.5	100,000	5,000
HBr	2000 ^f	10 ⁻²²	0.5	-	3
SO ₂	2050 ^f	10 ⁻²³	90	-	2

^a based on 2.5×10^{-5} detection sensitivity and 10 m optical path

^b ppm units

^c Fabry-Perot multimode lasers

^d delivered modules

^e AlGaAs

^f InGaAsP

Table 5. Gas Species That Can Be Monitored with a Single Laser Module.

Module Wavelength (nm)	Gas Species
750	NO ₂ , O ₂
1200	H ₂ O, HCl
1310	HF, H ₂ O, H ₂ S
1540	NH ₃ , C ₂ H ₂ , HCN, H ₂ O, CO ₂
1578	CO ₂ , CO, H ₂ S, H ₂ O
1650	CH ₄ , CO ₂ , C ₂ H ₄ , H ₂ O
1800	NO, HCl, H ₂ O

Along with the software upgrade, a few minor hardware modifications will be made.

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SUBJECT: Request Change in Distribution Statement

1. The U.S. Army Medical Research and Materiel Command has reexamined the need for the limitation assigned to technical reports written for Grant DAMD17-93-C-3085. Request the limited distribution statement for Accession Document Number ADB229302 be changed to "Approved for public release; distribution unlimited." This report should be released to the National Technical Information Service.

2. Point of contact for this request is Ms. Virginia Miller at DSN 343-7327 or by email at Virginia.Miller@det.amedd.army.mil.

FOR THE COMMANDER:

PHYLLIS M. RINEHART
Deputy Chief of Staff for
Information Management